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Chemical Determination of Heavy Metals in Pb and Zn Concentrates of Trepça (Kosovo) and Correlations Coefficients Study Between Chemical Data

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Original scientific paper

Fatbardh Gashi[1]; Stanislav Frančišković-Bilinski[2]*; Halka Bilinski[2]; Bashkim Thaçi[1]; Shkurta Shosholli[1]

[1] *Department of Chemistry, Faculty of Natural Sciences, University of Prishtina, Kosovo*

[2] *Institute "Ruđer Bošković", Division for marine and environmental research, POB 180, HR-10002 Zagreb, Croatia*

Abstract

Kosovo ore deposits are located in the Trepça belt which extends for over 80 km. The concentrate produced by the flotation process of the Trepça metallurgical corporation contains a considerable quantity of valuable metals, such as Pb, Zn, Fe and minor accompanying metals such as Cd, Cu, As, Sb, Bi, Ag, Au, etc. The subject of this work was to assess the concentration of major and minor metals in lead and zinc concentrates of Trepça and to study the correlation coefficients between metals. Chemical determination of concentrates was performed by using atomic absorption spectroscopy (AAS). In the content on lead concentrate samples, the following were found: Pb>Fe>Zn> Ag> As>Sb>Cd. In the content of zinc concentrate, the following were found: Zn>Fe>Pb>Ag>As>Cd. The program "Statistica ver. 6.0" has been used for calculations of basic statistical parameters, relationships between data and cluster analysis of R-mode. R-mode cluster analysis on lead concentrate samples showed that Pb has the closest linkages with Fe and they form one branch of the dendogram. On the zinc concentrate samples, Zn has the closest linkages with Fe and they form one branch of the dendogram.

Keywords

heavy metals, Pb and Zn concentrates, flotation process, atomic absorption spectrometry, Trepça

1. Introduction

Lead and zinc ores in the western Balkans region are extracted and are smelted since ancient Roman times. These ores, along with base metals, may also contain other metals (Cu, Ba, Ni, Bi, Cd), precious metals (Ag, Au) and the rare ones (In, Se, Te Ge, Ga, Ta, W, Va, etc.). The presence of metals Cu, Ba, Ni, Bi, Cd, Hg in ores, causes difficulty in further technological processing, but on the other hand, some of these metals (Bi, Cd, Cu, Ni, Ag, Au), if extracted, increase the economic value of the ore. Ore production and production of selective concentrates of Pb and Zn, is not in concordance with installed metallurgical capacities, and unavoidably will result in a high cost of treatment charges (**Azemi and Shyqri, 2014**). The flotation process is based on physico-chemical virtues of each mineral phase and the laws of their interaction. The flotation process is the selective separation of useful compounds from others. To reach a goal, the mining of the ore ingredient must remain in limbo in the water environment. At the same time, it is also distributed with air bubbles being sent in this aqua chemical environment. Selective extraction of minerals occurs when some of them attach to air bubbles and the rest remains in the water. The reaction of mineral particles with air bubbles is possible in those cases where these characteristics of hydrophobic particles have a natural ability or are created by the activity and impact of the chemical reagents (NaCN, $ZnSO_4$, Na_2CO_3, etc.) used in the flotation process (**Kawatra and Eisele, 1992; 2001**).

Trepça and the Stan Tërg deposit have been exploited since medieval times. It was mentioned for the first time in 1303 in the Dubrovnik Republic Chronicles (**Barié, 1977**). Trepça mines are a large industrial complex in Kosovo. Trep**ç**a was once one of the biggest companies in Yugoslavia. In the 1930s, a British company gained the rights to exploit the Stan Tërg mine close to Mitrovica city. Mines in Kosovo are categorized according to their geographic location: middle chain (Stan Tërg) and southern chain (Hajvalia, Artana and Kishnica). Lead and zinc mineralization in the Trepça belt is closely related to the NNW-SSE trending Vardar Zone, which marks the fundamental junction between the Serbo-Macedonian massif to the east and the Dinarides to the west (see **Figure 1**). This structure is characterized by Paleozoic crystalline schist's and phyllites overlain by Triassic clastics, phyllites and volcanoclastic rocks, Upper Triassic carbonates, Jurassic serpentinized ultrabasic rocks, gabbros, diabases and sediments of the ophiolite

Corresponding author: Stanislav Frančišković-Bilinski
francis@irb.hr

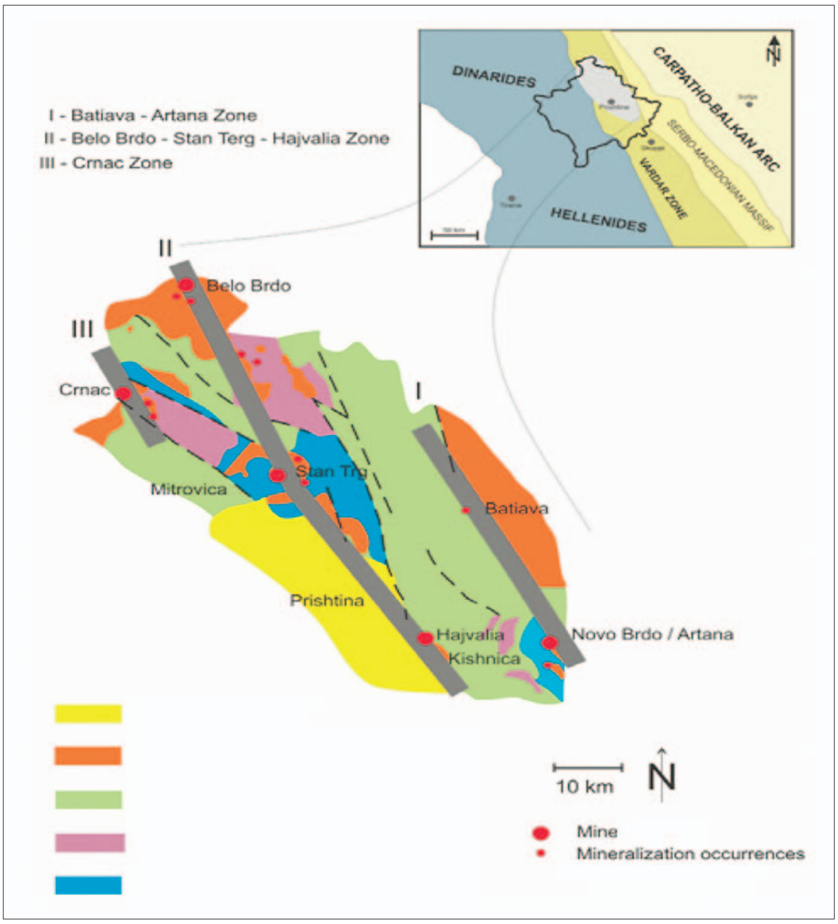

Figure 1: Geotectonic setting of Vardar zone and Trepça mineral belt units (**Hyseni et al., 2010**).

association, Cretaceous complex series of mélange and the Tertiary (Oligocene-Miocene) andesite, trachyte and latite subvolcanic intrusives, volcanics and pyroclastic rocks (**Miletic, 1997; Hyseni et al. 2010**). The Trepça belt is one of the largest Pb/Zn ore areas in Europe and extends for over 80 km in northern Kosovo and includes numerous lead and zinc deposits and occurrences (**Mineral deposits and mining districts of Serbia, 2002; Mining districts of Serbia, 2002**) The Trepça mineral belt (the Kopaonik metallogenic district) contains different types of Pb + Zn + Ag mineralization such as skarn, hydrothermal replacement and vein type of deposits (**Jankovič, 1995**). Ores are of various types: sulphide, sulphosalt and carbonate ones with quartz and tourmaline. The ores form numerous assemblages. For many minerals, more than one generation was established (**Smejkal, 1960**). The Pb-Zn mineralization is well positioned in Serbia, being particularly well concentrated in the Kopaonik district where the deposits form the backbone of the famous Trepça mining and metallurgical complex: Stani-Tërg, Belo Brdo, Novo Brdo, Hajvalija, etc. (**Mineral deposits, 2002**). The Trepça facility includes enterprises involved in mining, metallurgy, the chemical industry, metal processing and other types of production and manufacturing. Current primary products are ore and concentrates of lead and zinc with a significant content of silver and gold. Other products include sulphuric acid and zinc powder (**https://en.wikipedia.org/wiki/Trep%C4%8Da_Mines**). The Trepça ore is rich and contains 6 % of Pb, 4 % of Zn, 75 g/t of Ag and 102 g/t of Bi as well as several other elements including rare ones (**Féraud and Deschamps, 2009**).

Two types of ores from Stan Tërg: skarn and hydrothermal ore from Artana were investigated. Microscopic investigations in reflected light were performed. The chemical com-positions of some minerals were confirmed with an EDS microanalyser and will be followed up by further analytical work in the near future. The main ore minerals such as galena, sphalerite, arsenopyrite, pyrite and pyrrhotite were identified in Stan Tërg. The succession scheme for mineral precipitation in all samples is very similar. Chalcopyrite usually forms small minute blebs in sphalerite or in its inter-growth with galena and sphalerite. Sometimes, tetrahedrite is associated with it and forms small grains or veinlets in chalcopyrite. This type of tetrahedrite is poor in silver, its content of silver varying from 1 w % to 5 w % (**Ko£odziejczyk et al., 2012**).

Being associated with the industrial flotation process, **Haxhijaj et al., (2016)** have suggested that all balance sheet positions of zinc concentrate are variable. Zinc waste and losses in waste are of great value in the technological process of flotation, and to achieve minimum loss of Zinc in waste, we should check temperature flotation, grinding, classification, conditioning and drying. **Hyseni et al.** (**2011**) surveyed the distribution of the minor and accompanying elements such as Cu, As, Sb, Bi, Ni, Ca, Hg, Ti, Mn, Mo, Co and Mg. **Durmishaj et al.** (**2015**) have determined the contents of Pb and Zn in separate samples, whereas the content of Ag, Au, Cd, Bi, etc. were analyzed only as composite samples. The aim of the current work was to assess the concentration of major metals in Pb and Zn concentrates of ”Trepça” using Atomic absorption spectroscopy (AAS) and to perform correlations in the coefficients study between metals.

2. Materials and methods

Sampling was performed in the time period December 2014 – February 2015 and samples were taken in order to determine major/minor metals using AAS. Samples were analyzed in the laboratory of the same complex as well as in Department of Chemistry at the University of Prishtina for around three months. Metals in the aqua environment were analyzed using an atomic absorption spectrometer model “Perkin Elmer, AAS Analyst 400, HGA 900”. Accuracy of determination was ±10 %, and the emission line and limits of detection (LOD) of some metals are given in **Table 1**. The amount of water is determined at 110° C. All samples were digested in aqua regia (a mixture of HNO_3 and HCl = 1 : 3) and shaken for 1 h in room temperature for the determination of pseudo-total metal content (**ISO, 11466, 1995**). The sample dissolution procedure was performed by concentrated hydrochloric and nitric acids (suprapur, Merck). Single metal standard solutions of Pb, Fe, Zn, Ag, As, Sb and Cd (Merck, Germany) were used for the control and preparation of calibration standard solutions. All calibration standards were prepared by the appropriate dilution of standard stock solutions (1 g/L) in the range from 1 to 100 mg/L. All reagents that were used for the extraction procedure were of p.a. grade. The program “Statistica ver. 6.0” has been used for calculations of basic statistical parameters, relationships between data and cluster analysis of Q- and R- mode **(Statsoft, 2001; Tukey, 1977)**.

Table 1: Emission lines and limits of detection of some metals

Metal	λ /nm	LOD* /ppm
Pb	217.00	0.015
Fe	248.33	0.005
Zn	214.00	0.0015
Ag	328.00	0.0015
As	228.00	0.15
Sb	323.00	0.045
Cd	228.80	0.0008

*LOD: limit of detection.

3. Results

The average value of metals on Pb and Zn concentrate samples (obtained by the AAS method) are presented in **Tables 2 and 3**. The descriptive statistics summaries of the selected variables of samples are presented in **Tables 4 and 6**. For each variable, the values were given as an arithmetic mean, geometric mean, median, minimal and

Table 2: Percentage of Pb, Fe, Zn, Ag, As, Sb and Cd in lead concentrate samples

	Pb (w %) (Mean)	Fe (w %) (Mean)	Zn (w %) (Mean)	Ag (w %) (Mean)	As (w %) (Mean)	Sb (w %) (Mean)	Cd (w %) (Mean)
Sample 1	67.77	6.25	0.98	0.099	0.21	0.16	0.011
Sample 2	61.57	7.01	1.25	0.091	0.17	0.19	0.090
Sample 3	59.34	7.55	1.46	0.084	0.18	0.21	0.012
Sample 4	59.86	7.66	1.32	0.0835	0.11	0.16	0.016
Sample 5	57.93	7.15	1.37	0.087	0.20	0.18	0.010

Table 3: Percentage of Zn, Fe, Pb, Ag, As and Cd in zinc concentrate samples

	Zn (w %) (Mean)	Fe (w %) (Mean)	Pb (w %) (Mean)	Ag (w %) (Mean)	As (w %) (Mean)	Cd (w. %) (Mean)
Sample 1	48.38	13.05	1.26	0.00282	0.08	0.20
Sample 2	48.61	12.41	1.21	0.00245	0.15	0.35
Sample 3	49.71	11.50	1.80	0.002735	0.11	0.28
Sample 4	47.32	12.75	1.19	0.002624	0.08	0.32
Sample 5	48.50	12.73	1.24	0.002745	0.10	0.27

Table 4: Basic statistical parameters of metals in lead concentrate samples

Variable (w %)	Descriptive Statistics						
	Mean	Geometric	Median	Minimum	Maximum	Variance	Std. Dev.
Pb	61.29400	61.20105	59.86000	57.93000	67.77000	14.80143	3.847263
Fe	7.12400	7.10585	7.15000	6.25000	7.66000	0.31158	0.558194
Zn	1.27600	1.26461	1.32000	0.98000	1.46000	0.03323	0.182291
Ag	0.08890	0.08872	0.08700	0.08350	0.09900	0.00004	0.006387
As	0.17400	0.16985	0.18000	0.11000	0.21000	0.00153	0.039115
Sb	0.18000	0.17902	0.18000	0.16000	0.21000	0.00045	0.021213
Cd	0.02780	0.01802	0.01200	0.01000	0.09000	0.00121	0.034845

Table 5: The correlation factor of analytical variables in lead concentrate samples

Variable	Correlations, marked correlations are significant at $p < .05000$						
	Pb	Fe	Zn	Ag	As	Sb	Cd
Pb	1.00						
Fe	-0.86	1.00					
Zn	-0.95	0.90	1.00				
Ag	0.91	-0.99	-0.94	1.00			
As	0.37	-0.71	-0.34	0.62	1.00		
Sb	-0.48	0.39	0.67	-0.41	0.21	1.00	
Cd	0.03	-0.08	-0.07	0.15	-0.12	0.24	1.00

maximal concentration, variance and standard deviation. The correlation Pearson's factor for 7 variables were calculated to see if some of the parameters were interrelated with each other and the results are presented in **Tables 5 and 7**. Cluster means of variables obtained using the R-mode cluster analysis (dendograms of spatial clustering for measured variables) are presented in **Figures 2 and 3**.

4. Discussion

4.1. Discussion of metal concentrations on lead/zinc concentrate

Aqua regia digestion was used to determine metals (Pb, Fe, Zn, Ag, As, Sb and Cd) in lead and zinc concentrate samples (see **Tables 2 and 3**). In the content of lead concentrate samples, the following metals were found: Pb 57.93-67.77 % with a mean value ± standard deviation from 61.294% ±3.8473, Fe 6.25-7.66% with a mean value ± standard deviation from 7.124 % ±0.5582, Zn 0.98-1.46 % with a mean value ± standard deviation from 1.276% ±0.1823, Ag 0.0835-0.099% with a mean

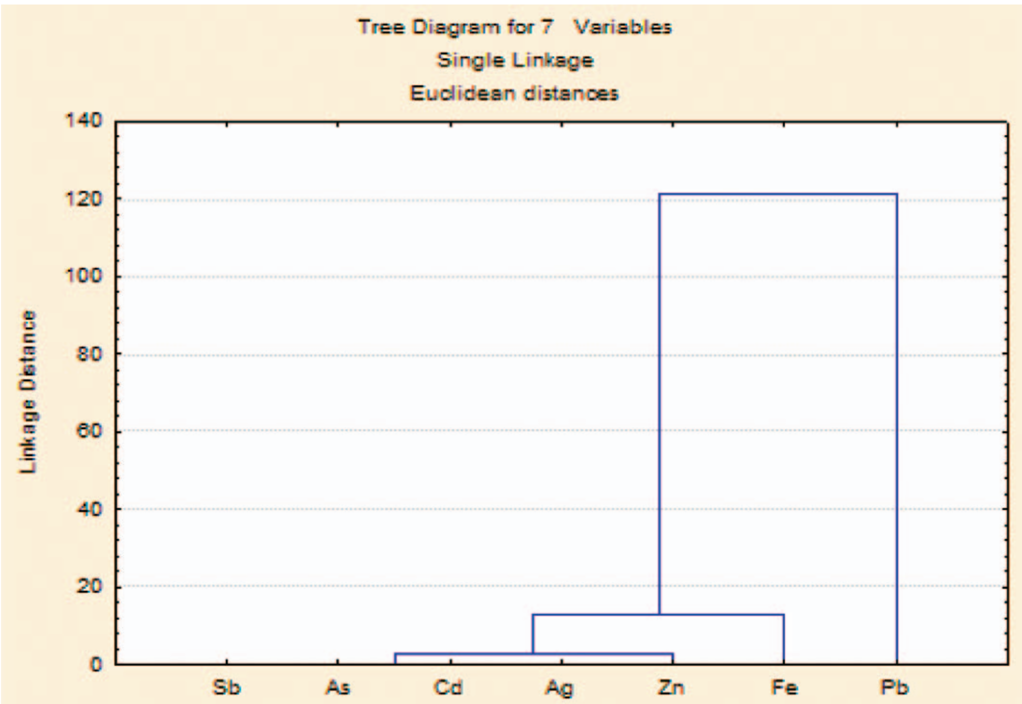

Figure 2: Cluster analysis R-mode of some measured variables in lead concentrate samples.

Table 6: Basic statistical parameters of metals in zinc concentrate samples

Variable (w %)	Descriptive Statistics						
	Mean	**Geometric**	**Median**	**Minimum**	**Maximum**	**Variance**	**Std. Dev.**
Zn	48.50400	48.49806	48.50000	47.32000	49.71000	0.720730	0.848958
Fe	12.48800	12.47624	12.73000	11.50000	13.05000	0.356320	0.596925
Pb	1.34000	1.32276	1.24000	1.19000	1.80000	0.066850	0.258554
Ag	0.00267	0.00267	0.00274	0.00245	0.00282	0.000000	0.000144
As	0.10400	0.10110	0.10000	0.08000	0.15000	0.000830	0.028810
Cd	0.28400	0.27910	0.28000	0.20000	0.35000	0.003230	0.056833

Table 7: The correlation factor of analytical variables in zinc concentrate

Varable	Correlations, marked correlations are significant at p <.05000, N=5					
	Zn	**Fe**	**Pb**	**Ag**	**As**	**Cd**
Zn	1.00					
Fe	-0.78	1.00				
Pb	0.83	-0.90	1.00			
Ag	0.19	0.13	0.31	1.00		
As	0.44	-0.44	0.09	-0.76	1.00	
Cd	-0.16	-0.31	-0.13	-0.92	0.64	1.00

value ± standard deviation from 0.0889 % ±0.0064, As 0.11-0.21% with amean value ± standard deviation from 0.174% ±0.0391, Sb 0.16-0.21% with a mean value ± standard deviation from 0.18%±0.0212 and Cd 0.01-0.09% with a mean value ± standard deviation from 0.0278%±0.0348.

The content of zinc concentrate samples were found: Zn 47.32-49.71% with a mean value ± standard deviation from 48.504% ±0849, Fe 11.50-13.05% with a mean value ± standard deviation from 12.488% ±0.597, Pb 1.19-1.80% with a mean value ± standard deviation from 1.34% ±0.258, Ag 0.00245-0.00282% with a mean value ± standard deviation from 0.00267% ±0.000144, As 0.08-0.15% with a mean value ± standard deviation from 0.104% ±0.02881 and Cd 0.2-0.35% with a mean value ± standard deviation from 0.284%±0.0568.

4.2. Statistical interpretation of results

a) Basic statistical parameters (Mean, Geometric mean, Median, Minimum, Maximum, Variance and Standard deviation) for 7 parameters analyzed in 5 samples are presented in **Tables 4 and 6**.

b) The statistical regression analysis has been found a highly useful technique for the linear correlation between various water parameters. The correlation coeffi-

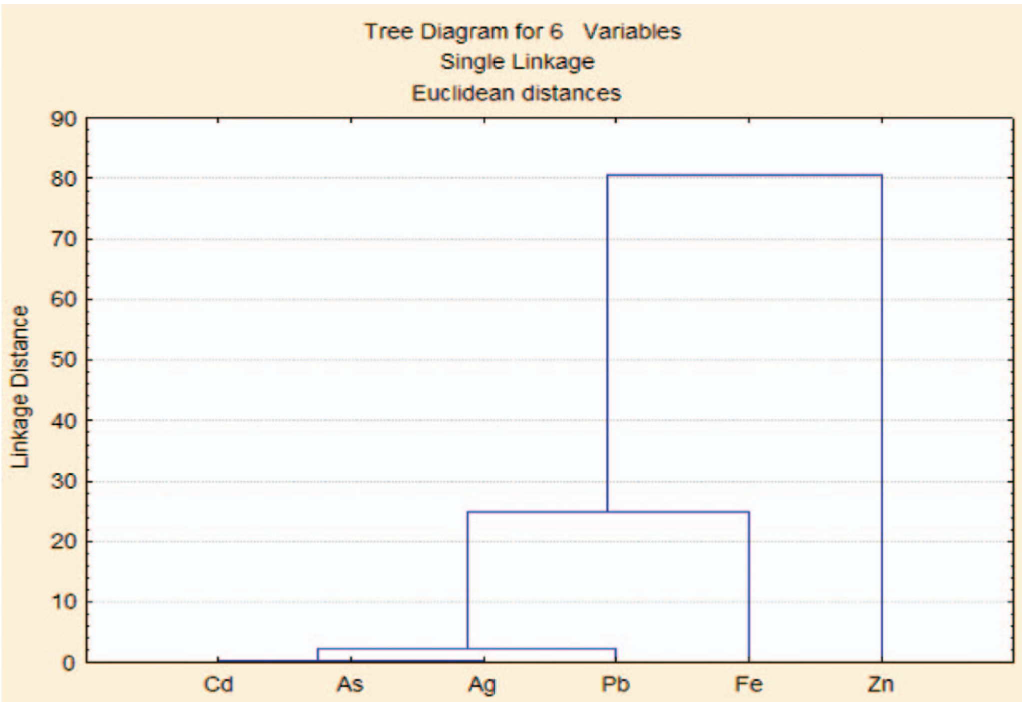

Figure 3: Cluster analysis R-mode of some measured variables in zinc concentrate samples.

cient indicates positive and negative significant correlation of variables with each other. Positive correlation means one parameter increases with other parameters and negative correlation means one parameter increase while other parameters decrease. Based on the statistical regression analysis of lead concentrate samples (see **Table 5**), the result suggests a very strong Pearson correlation factor of Pb with Ag (0.91), which could be associated to galena with pyrargyrite as well as with the isomorphic enrichment of galena with Ag. Also, the result values suggest a correlation (Pearson correlation factor 0.37) of Pb with As, which could be related to the association of galena with arsenopyrite (**www.mindat.org/min-305.html**) as well as with the isomorphic enrichment of galena with As. Result values also suggest a very strong correlation of Fe with Zn (0.90) and a strong correlation with Sb (0.39); Zn is strongly correlated with Sb (0.67); As is strongly correlated with Sb (0.21) and Sb is strongly correlated with Cd (0.24). Based on the statistical analysis of zinc concentrate samples (see **Table 7**), the results suggest a very strong Pearson correlation factor of Zn with Pb (0.83), which could be associated with sphalerite (**www.minerals.net/mineral/sphalerite.aspx**) with galena as well as with the isomorphic enrichment of sphalerite and galena with A, also Zn correlated with As (0.44) and Ag (0.19); Fe is correlated with Ag (0.13); Pb is correlated with Ag (0.31) and As (00.09) and As is correlated with Cd (0.64).

c) Cluster analysis of R-mode (see **Figures 3 and 4**) showed mutual links between the studied parameters. It could be observed on lead concentrate samples that Pb has the closest association with Fe and they form one branch of the dendogram. This branch is linked with the other one, in which Zn, Ag and Cd are linked, after which As and Sb follow. On the zinc concentrate samples, Zn has the closest association with Fe and they form one branch of the dendogram. This branch is linked with the other one, in which Pb, Ag and As are linked, after which Cd metal follows.

5. Conclusions

The Trepça mines are rich in polymetallic minerals. The results showed that the percentage of lead, zinc and iron in all concentrate samples were very high and lower percentage of silver, arsenic, antimony and cadmium were found. In the content of lead concentrate samples, the following were found (mean value± standard deviation): Pb (61.294% ±3.8473), Fe (7.124% ±0.5582), Zn (1.276% ±0.1823), Ag (0.0889% ±0.0064), As (0.174% ±0.0391), Sb (0.18%±0.0212) and Cd (0.0278%±0.0348). In the content of zinc concentrate samples, the following were found (mean value ± standard deviation): Zn (48.504% ±0849), Fe (12.488% ±0.597), Pb (1.34% ±0.258), Ag (0.00267% ±0.000144), As (0.104% ±0.02881) and Cd (0.284%±0.0568).

The statistical regression analysis has shown a high positive correlation between Pb and Ag, which could be the association of galena with pyrargyrite as well as with the isomorphic enrichment of galena with Ag. Also, the result values suggest a correlation of Pb with As, which could be related to the association of galena with arsenopyrite as well as with the isomorphic enrichment of galena with As. Result values also suggest a very strong correlation of Fe with Zn and Sb; Zn is strongly correlated with Sb; As is strongly correlated with Sb and Sb is strongly correlated with Cd. Based on the statistical analysis of zinc concentrate samples, the results suggest a very strong correlation of Zn with Pb, which could be the association of sphalerite with galena as well as with the isomorphic enrichment of sphalerite and galena with A, also Zn correlated with As and Ag; Fe was correlated with Ag; Pb was correlated with Ag and As and As was correlated with Cd. R-mode cluster analysis showed in lead concentrate samples that Pb has the closest association with Fe and they form one branch of the dendogram. In the zinc concentrate samples, Zn has closest association with Fe and they form one branch of the dendogram.

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SAŽETAK

Kemijsko određivanje teških metala na koncentratima olova i cinka u Trepçi (Kosovo) i studija korelacijskih koeficijenata između kemijskih podataka

Kosovski rudni depoziti nalaze se u pojasu Trepçe, koji se prostire u duljini više od 80 km. Koncentrat proizveden u flotacijskome procesu metalurške korporacije Trepça sadržava znatnu količinu vrijednih metala, kao što su Pb, Zn, Fe, i manje količine pratećih metala, kao što su Cd, Cu, As, Sb, Bi, Ag, Au itd. Predmet ovoga rada bio je ispitati koncentracije metala u koncentratima olova i cinka u Trepçi i odrediti koeficijente korelacije između metala. Karakterizacija ispitanih uzoraka provedena je upotrebom atomske adsorpcijske spektroskopije (AAS). U sadržaju uzoraka olovnih koncentrata pronađeni su: Pb, Fe, Zn, Ag, As, Sb, Cd. U sadržaju koncentrata cinka pronađeni su: Zn, Fe, Pb, Ag, As, Cd. Program „Statistica ver. 6,0" korišten je za izračun osnovnih statističkih parametara, korelacijskih koeficijenata između podataka i klasterske analize R-modaliteta. Klasterska analiza R-modaliteta na koncentratima olova pokazala je da Pb ima najbliže veze s Fe, tako da zajedno čine jednu granu dendograma. Na uzorcima koncentrata cinka Zn ima najbliže veze s Fe, tako da zajedno čine jednu granu dendograma.

Ključne riječi

teški metali, koncentrati olova i cinka, flotacija, atomska adsorpcijska spektrometrija, Trepça